

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problems Mailbox.**

PATENT SPECIFICATION

(11) 1 333 475

1 333 475

NO DRAWINGS

(21) Application No. 13493/71 (22) Filed 6 May 1971

(31) Convention Application No. P 20 24 051.1

(32) Filed 16 May 1970 in

(33) Germany (DT)

(44) Complete Specification published 10 Oct. 1973

(51) International Classification A61K 7/00; C11D 1/74

(52) Index at acceptance

ASB 771 774

CSD 6B11A 6B11C 6B12B1 6B12F1 6B12F2 6B12L

6B12N4 6B12NX 6B4 6C8



(54) COSMETIC COMPOSITIONS

- (71) We, HENKEL & CIE, GMBH, a German Company, of 67, Henkelstrasse, Duesseldorf, 4000, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- The invention relates to a composition for use in cosmetic preparations, especially cosmetic cleansing agents, containing substances for replacing the oil in the skin, based on esterification products of glycerine-ethylene oxide adducts with long-chain fatty acids.
- Cosmetic cleansing agents such as shampoos, foam baths, toilet soaps and similar products cause a more or less great removal of oil from the skin on repeated use. This phenomenon is particularly pronounced when the cleansing agents are based on synthetic, surface-active substances such as alkylbenzene sulphonates, fatty alcohol sulphates, olefine sulphonates, fatty alcohol ether sulphates and other surface-active compounds. Attempts have therefore been made to neutralise this removal of oil from the skin by replacing the oil by means of suitable additions to the cleansing agents. Allowance has in such case to be made for disadvantages, however, since the products generally concerned in replacing the oil in the skin have an unfavourable action on the foaming properties of the cleansing agents and, in cosmetic preparations based on alcoholwater mixtures, show insufficient solubility.
- The present invention provides a composition for use in cosmetic preparation which comprises a surface active compound and the esterification product of an ethylene oxide addition compound produced from glycerine and 4 to 20 mol of ethylene oxide per mol of glycerine, with a fatty acid of chain length from 8 to 18 carbon atoms in a ratio of 1 to 2 mol of fatty acid to 1 mol of glycerine-ethylene oxide addition compound as oil replacement material.
- Esterification products of ethylene oxide addition compounds produced from glycerine and 7 to 15 mol of ethylene oxide per mol of glycerine with fatty acids of chain length from 8 to 18 carbon atoms in a ratio of 1 mol of fatty acid to 1 mol of glycerine-ethylene oxide addition compound are preferred as the oil replacement materials.
- The preparation of the ethylene oxide addition compound as intermediate product was generally effected in known way by reacting glycerine with ethylene oxide in the desired proportions with alkaline catalysis by means of sodium ethylate. For the further treatment, the ethylene oxide addition compound obtained was reacted in the usual way with a fatty acid of chain length from 8 to 18 carbon atoms in the molar ratio of 1:1 or 1:2, using isopropyl titanate as esterification catalyst. The esterification products obtained were light-coloured to yellowish liquids of low viscosity with an oil character to lard-like products of a faint self colour.
- The quantities of oil replacement material according to the invention used in the cosmetic preparations may vary within very wide limits according to the product and its oil-removing action, and generally vary from 2 to 50% by weight, especially 5 to 25% by weight. Still higher additions are possible if the esterification products according to the invention are used at the same time in their property as surface-active substances, but in most cases this use will be of small advantage.
- Oil replacement materials to be used according to the invention include, for example, esterification products from

			the adduct of 1 mol of glycerine + 4 mol of ethylene oxide with 1 mol of coconut fatty acid C_{8-18}
		" "	+ 6 mol of ethylene oxide with 2 mol of coconut fatty acid C_{8-18}
5		" "	+ 7 mol of ethylene oxide with 1 mol of coconut fatty acid C_{8-18}
		" "	+ 7 mol of ethylene oxide with 1 mol of tallow fatty acid
10		" "	+ 8 mol of ethylene oxide with 1 mol of oleic acid
		" "	+ 9 mol of ethylene oxide with 1 mol of palm kernel fatty acid
		" "	+ 10 mol of ethylene oxide with 1 mol of tallow fatty acid
15		" "	+ 10 mol of ethylene oxide with 2 mol of groundnut oil fatty acid
		" "	+ 12 mol of ethylene oxide with 1 mol of coconut fatty acid C_{8-18}
20		" "	+ 15 mol of ethylene oxide with 1 mol of palm kernel fatty acid
		" "	+ 15 mol of ethylene oxide with 2 mol of tallow fatty acid

The present invention will be further described by way of illustration with reference to the following examples. Abbreviations and units used in the examples are defined as follows:—

"Acid value" is the number of mg of potassium hydroxide which are needed to neutralize the free esterified fatty acid contained in 1 g of substance;

"Saponification value" is the number of mg of potassium hydroxide which are needed to completely saponify 1 g of ester;

35 "Hydroxyl number" is the number of mg of potassium hydroxide which are needed to neutralize the acetic acid which is absorbed by 1 g of material. The procedure used is as follows; the substance being investigated is weighed out exactly and is then acetylated by heating with crystalline acetic acid, all the OH groups present thereby being esterified. Subsequently, the acetylated portion of the material is separated by a shaking and washing procedure and the acetylated part is saponified using boiling potassium hydroxide solution. The excess potassium hydroxide which is present after the saponification steps is determined by titration with hydrochloric acid.

"EO" is the ethylene oxide group;
"WAS" is active washing substance.

All parts used throughout the examples are by weight unless otherwise specified.

Examples

The following esterification products were used for the experiments and cosmetic preparations described below.

- (A) (1 mol of glycerine + 7.4 mol of ethylene oxide) with 1 mol of coconut fatty acid C_{8-18} 60
Acid value 1.0, sap. value 92, hydroxy value 185
- (B) (1 mol glycerine + 7.4 mol of ethylene oxide) with 1 mol of tallow fatty acid 65
(Acid value 1.1, sap. value 83, hydroxy value 166
- (C) (1 mol glycerine + 10 mol of ethylene oxide) with 1 mol of tallow fatty acid 70
Acid value 1.4, sap. value 71, hydroxy value 141

Since for cosmetic cleansing compositions the ability to combine with certain surface-active compounds is of essential importance, mixtures given in the following Table were tested.

TABLE I

Mixing component	Mixture 1	Mixture 2	Mixture 3
(A)	10	—	—
(B)	—	10	—
(C)	—	—	10
Sodium lauryl ether sulphate (2 EO) (27—28% WAS)	50	50	50
Water	40	40	40
Result	Clear homo-geneous solution	Clear homo-geneous solution	Clear homo-geneous solution

In a further experiment the foaming power of a foam bath basic recipe with additions of the individual oil replacement materials was examined.

TABLE II

Components	Mixture 1	Mixture 2	Mixture 3	Mixture 4	Mixture 5
Sodium lauryl ether sulphate (2 EO) (27—28% WAS)	60	60	60	60	60
Sodium lauryl sulphate (over 90% WAS)	5	5	5	5	5
Isopropyl myristate	—	5	—	—	—
(A)	—	—	5	—	—
(B)	—	—	—	5	—
(C)	—	—	—	—	5
Water	35	30	30	30	30
Appearance	clear	turbid deposit formed	clear	clear	clear
Foaming power					
Initial volume in ml 1 minute after beating					
0.5 g/litre	260	170	300	340	240
1.0 g/litre	510	250	490	490	400
2.0 g/litre	660	330	610	640	540
Breaking down of the volume of foam in ml/minute					
0.5 g/litre	2.0	2.5	4.0	4.5	1.5
1.0 g/litre	5.5	3.5	7.0	8.0	4.0
2.0 g/litre	8.0	6.5	9.5	3.0	6.5

5 The foaming power of the individual mixtures was measured in the foam-beating machine according to DIN (Deutsche Industrie Norm) classification No. 53,902, in which the volume of foam was measured. The figures for the foam were taken at 45°C in water of 10° German hardness after 30 beats. The measurement was taken 1 minute after the end of the beating and 21 minutes after the end of the beating. The breakdown of the volume of foam was calculated in ml/minute from the decrease of the volume of foam in

20 minutes. The amounts given in g/litre relate to the respective mixture.

As may be seen from the above Table, the foam values of the mixtures containing oil replacement means according to the invention are substantially better than when isopropyl myristate is used as oil replacement materials, and are scarcely inferior to the figures for a mixture of pure detergent substances.

A few formulations for cosmetic preparations containing oil replacement materials according to the invention are given below.

15

20

25

Clear Shampoo

Sodium lauryl sulphate (2EO) (27—28% WAS)	40	parts by weight		
Coconut fatty acid diethanolamide	6	"	"	"
Oil replacement means (A)	10	"	"	"
Water	44	"	"	"

Shampoo for dry hair

Sodium lauryl ether sulphate (2 EO) (27—28% WAS)	20	parts by weight		
Sodium lauryl sulphate (90% WAS)	5	"	"	"
Coconut fatty acid diethanolamide	3	"	"	"
Coconut fatty acid monoethanol- amide paste 30%	5	"	"	"
Water-soluble vitamin F	0.5	"	"	"
Oil replacement means (B)	25.0	"	"	"
Water	41.5	"	"	"

Foam bath

Sodium lauryl ether sulphate (2 EO) (27—28% WAS)	30	parts by weight		
Sodium lauryl sulphate (90% WAS)	15	"	"	"
Coconut fatty acid diethanolamide	5	"	"	"
Pine-needle oil	5	"	"	"
Oil replacement means (C)	10	"	"	"
Water	35	"	"	"

Hair wash

Isopropanol	60.0	parts by weight		
Menthol	0.2	"	"	"
Calcium pantothenate	0.05	"	"	"
Vitamin H	0.30	"	"	"
Inositol	0.10	"	"	"
Perfume	0.50	"	"	"
Oil replacement means (C)	5.00	"	"	"
Water	33.85	"	"	"

After-shave lotion

Ethyl alcohol 96%	65.0	parts by weight		
Menthol	0.2	"	"	"
Camphor	0.2	"	"	"
Peruvian balsam	0.1	"	"	"
Perfume	0.5	"	"	"
Glycerine	5.0	"	"	"
Witch hazel extract	10.0	"	"	"
Boric acid	0.5	"	"	"
Oil replacement means (A)	10.0	"	"	"
Water	8.5	"	"	"

Sun tan cream

Colloidally dispersed mixture of 90 parts of cetylstearyl alcohol and 10 parts of sodium lauryl sulphate

2-Octyldodecanol

Groundnut oil

Light-protective means

Oil replacement means (B)

Water

10.0 parts by weight

10

" " "

5

" " "

2

" " "

20

" " "

53

" " "

The oil replacement compositions according to the invention can be used particularly advantageously in cosmetic cleansing means, because they do not exert any appreciable influence on the foaming power of the surface-active products, and because they already have a good solubility in alcohol-water mixtures.

10 WHAT WE CLAIM IS:—

1. A composition, for use in cosmetic preparations, which comprises a surface active compound and the esterification product of an ethylene oxide addition compound produced from glycerine and 4 to 20 mol of ethylene oxide per mol of glycerine with a fatty acid of chain length from 8 to 18 carbon atoms in a ratio of 1 to 2 mol of fatty acid to 1 mol of glycerine-ethylene oxide addition compound as oil replacement material.

2. A composition according to claim 1, in which the ratio of glycerine to ethylene oxide in the addition compound is from 7 to 15 mols

of ethylene oxide per mol of glycerine.

3. A composition according to claim 1 or 2 wherein the fatty acids of chain length from 8 to 18 carbon atoms are in a ratio of 1 mol of fatty acid to 1 mol of the glycerine-ethylene oxide addition compound. 25

4. A composition according to claims 1 to 3, containing from 2 to 50% by weight of the oil replacement material. 30

5. A composition according to claim 1 to 4 containing from 5 to 25% by weight of the oil replacement material. 35

6. A composition according to claim 1 substantially as hereinbefore described with reference to and as illustrated in the foregoing examples.

7. A cosmetic preparation whenever containing a composition as claimed in any one of claims 1 to 6. 40

W. P. THOMPSON & CO.,
12, Church Street, Liverpool, L1 3AB.
Chartered Patent Agents.